GRScientific

Aquamax KF *Oil Evaporator*

User Manual

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Chapter 1 Introduction

1.1 Instrument Description

Key personnel at G.R. Scientific are recognised experts in Coulometric Karl Fischer titration technology and electrochemistry. They have designed and manufactured Coulometric titrators since the early 1980's and are widely regarded as some of the leading specialists in this technique.

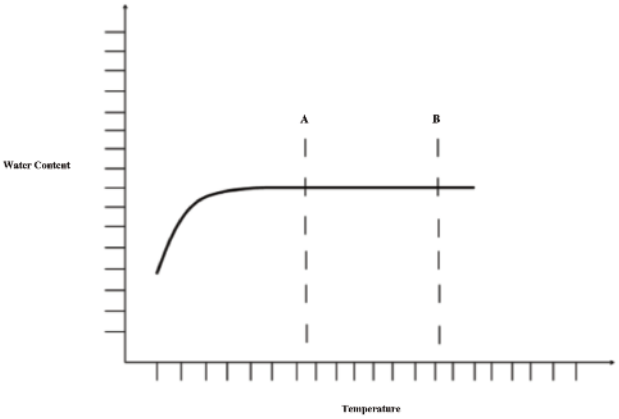
The Aquamax KF Oil Evaporator model 510000 has been designed as a "stand-alone" unit so that it can be used in conjunction with most leading models of Coulometric and volumetric Karl Fischer titrators and to comply with the requirements of ASTM method D6304, "Standard test method for determination of water in petroleum products, lubricating oils and additives by Coulometric Karl Fischer titration".

1.2 Principle of Measurement

Karl Fischer titration is simply a means to measure water content of samples. Modern instruments, such as the Aquamax KF, use the Coulometric principle. This is an absolute technique which does not require calibration of the reagents. For further information, visit www.grscientific.com.

It is important to know the water content of lubricating oils, additives and similar samples to enable accurate prediction of performance and quality. The presence of water in such samples could lead to corrosion and wear of machinery and reduce the effectiveness of the lubricating additives.

If you are analysing an unknown sample then we suggest plotting a graph of water content against temperature as per following example. This will enable you to determine the best temperature range for a sample



The two main parameters that you can change are gas flow rate and temperature. We suggest using carrier gas flow rate of 0.30 litre per minute (300 ml/min).

Oven temperatures are usually set between 120 – 150°C. The maximum selectable temperature is 200°C and there is a thermal cut-out control above this temperature.

For most lube oils we would suggest flow rate of 0.3 litres per minute and temperature of 140°C.

1.3 Instrument Connections

Various ports are located on the back plate of the instrument. Below is a list of ports that can be found:

(1) Power Supply Port

The power supply port is for connecting to a 24V mains power supply, using the supplied power adaptor.

(2) Thermocouple Port

This port is for connecting the thermocouple.

(3) Gas Out Port

This port is for connecting the tubing to the desiccant bottle for when the gas is released through the tubing.

(4) Gas In Port

The gas in port is for connecting a carrier Gas supply.

Chapter 2 Safety Information

2.1 General Safety Points

GR Scientific products are in conformity with the EU Directives 2004/108/EC and 2006/95/EC. This is based upon the compliance of the products with the harmonised standards: BS EN 61000-4-2:2009; BS EN 61010-2-030:2010; and BS EN 61326-1:2013.

GR Scientific products are manufactured in accordance to RoHS following the EU Directive 2011/65/EU; it does not contain any of the restricted substances in concentrations and applications banned by this directive.

2.2 Electrical Safety

The Aquamax KF Oil Evaporator complies with the international standard IEC 61010.

Only operate this instrument within the mains voltage supply that is specified on the back the instrument, an incorrect mains supply can damage the instrument. Never open the housing of the instrument, only GR Scientific personnel who are trained to service the instrument can open the housing.

There is risk of electrocution if live components are touched.

Always ensure the instrument is disconnected from the mains power supply when connecting or disconnecting cables from the back of the instrument, as electrostatic discharge can build up.

2.3 Tubing and Connections

Leaks in the tubing are a safety risk if left without attention. Ensure that all tubing is screwed into the connections tightly by hand, without excessive force. Breaks in the tubing connections or on the tubing can lead to spillages; check the connections regularly to avoid this.

2.4 Solvent and Chemical Safety

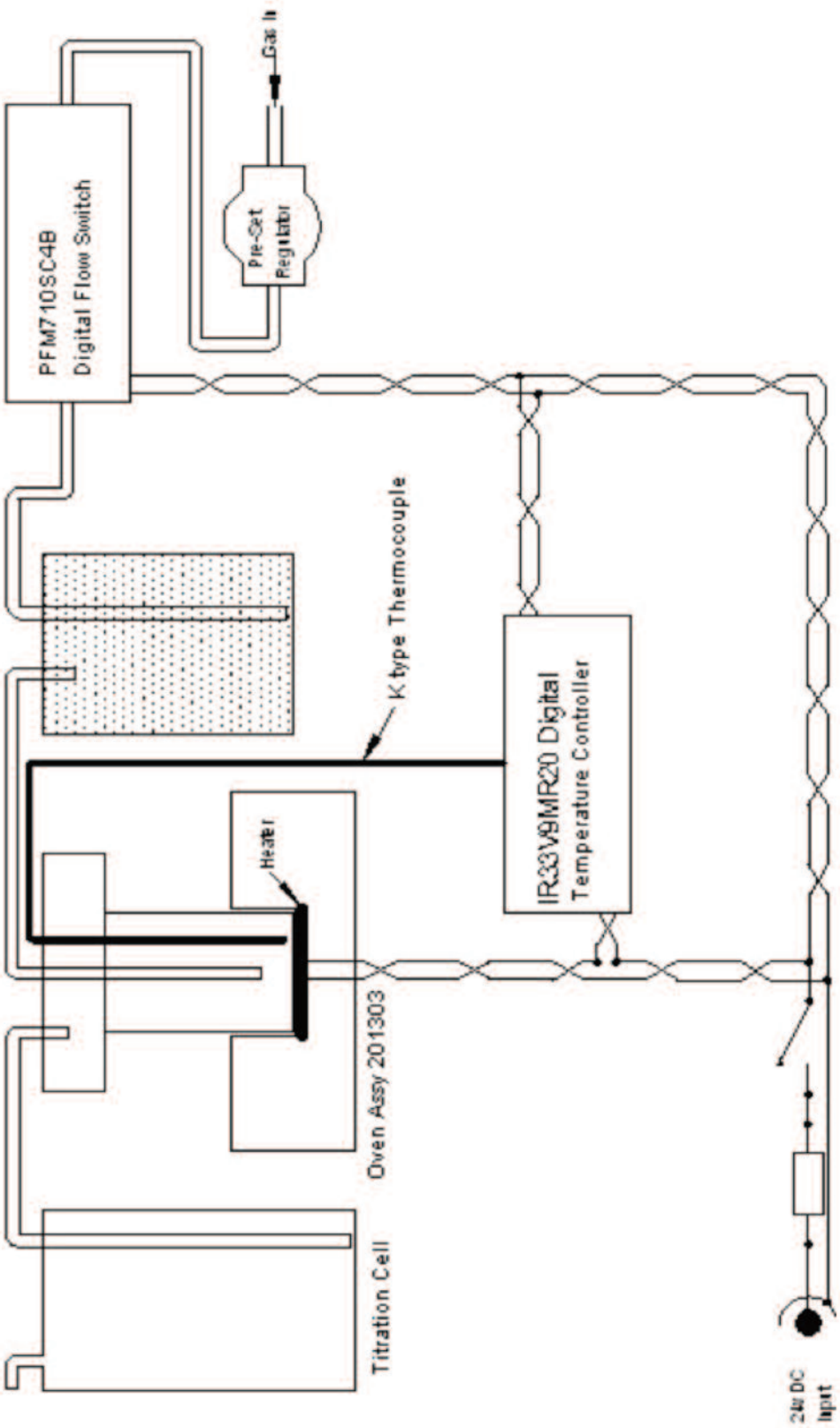
When using flammable or irritant solvents and chemicals, follow suitable safety measures to protect yourself and others around. Always use the instrument in a well ventilated area, clean up spilled fluids immediately, and keep flame sources away from the work area. Always follow the safety instructions of the chemical manufacturer and apply the correct countermeasures that are required.

2.5 Recycling and Disposal

This instrument is covered by the WEEE (Waste Electronic and Electrical Equipment) Directive 2012/19/EU to prevent the negative and harmful effects on the environment and public health. For more information on how to dispose of the instrument safely, please contact your local authority, your local waste disposal company or GR Scientific directly.

Chapter 3 Instrument Overview

3.1 System Schematic Overview



3.2 Front Panel

(1) Carrier Gas from Desiccant Bottle
(2) Thermocouple
(3) Temperature Meter
(4) Gas Flow Meter

3.3 Rear Panel

(1) Thermocouple Port
(2) Gas Out Port
(3) Gas In Port
(4) I/O Switch
(5) Fuse
(6) Power Input
(7) Bottle Clamp

Chapter 4 Instrument Set Up

4.1 Receiving an Aquamax KF Oil Evaporator

4.1.1 Packaging

The Aquamax KF Oil Evaporator is packaged in suitable protective packaging to ensure that no damage occurs during transit, only this packaging is suitable for the safe transportation during transit. Please keep this packaging as transit of the instrument may need to occur in the future.

4.1.2 Checks

Once you have received the instrument, immediately check to make sure that there is no damage and no parts are missing. A delivery note will be included within the package to use to compare with the shipment.

Ensure that inside the shipment has a calibration certificate for the instrument, if not please request this from the manufacturer.

4.1.3 Location

The Aquamax KF Oil Evaporator has been developed for indoor use only. Locate the instrument within the work area where it is free from excessive:

- vibrations
- direct sunlight
- contamination from chemicals
- explosive environments
- corrosive atmospheres
- major temperature fluctuations

4.2 Connecting the Power Supply

The Aquamax KF Oil Evaporator is powered by an external mains source, with a power pack to ensure this connection is the correct voltage of 24V DC for the instrument.

Damage may occur beyond repair and the user may be suspect to electric static shocks if the incorrect power is supplied.

Note: Refer to Chapter 2 for more safety information.

To avoid risk of electrocution, make sure the I/O switch is set to OFF before connecting the power supply.

4.3 Connecting to an Aquamax KF Coulometric

(1) Fit gas inlet nipple to rear of evaporator.

(2) Locate small “O” rings and injection septa inside holes of vessel lid and screw in compression fittings. Do not over-tighten as other items will need to be fitted.

(3) Connect long length tubing to gas distribution tube, feed through correct compression fitting and connect to desiccant bottle outlet fitting. Gas distribution tube should be located at bottom of vessel to allow optimum bubbling of carrier gas through the base oil and samples. Apply smear of silicone grease to large“O” ring of lid assembly before locating onto evaporator glass vessel. Tighten compression fitting.

(4) Position thermocouple in vessel lid and tighten compression fitting. Connect cable to rear panel of evaporator. Position thermocouple so that it is half way between bottom of vessel and the line indicating base oil depth. Do not locate so that it is touching bottom of vessel.

(5) Connect short length tubing to the inside of desiccant bottle lid. This should be from carrier gas supply cylinder so that gas flows through maximum amount of desiccant. Fill desiccant bottle with molecular sieve. Locate filled desiccant bottle on rear of evaporator unit. Outlet of desiccant bottle should be connected to the gas distribution tube.

(6) Using a funnel, measuring cylinder or suitable volume syringe, add 10ml base oil to evaporator vessel.

(7) Connect gas sample inlet and outlet adapters to Aquamax KF titration vessel.

(8) Charge the titration vessel with 150ml anode and 10ml cathode reagents and allow to precondition as per titrator user manual instructions. These larger volumes of reagents allow greater depth of reagent for the carrier gas to bubble through.

(9) Connect the outlet from vessel of oil evaporator to gas inlet adapter of titration vessel using short length tubing and elbow connector. This distance between evaporator and titrator should be kept to minimum to reduce any problems of condensation in transfer tubing.

(10) Set oven temperature.

(11) Turn three way cock of gas inlet adapter so that gas flow is directed away from titration vessel out of the side arm. This allows drying of all carrier gas lines up to the connection with titrator.

(12) Set gas flow rate.

(13) Vent the outlet adapter from titration vessel to fume hood or outdoors using some plastic tubing or similar.

(14) After the gas lines have been sufficiently dried, divert the gas flow, using the three way cock, so that it bubbles through the titration vessel. The background drift value of the titrator will increase whilst the gas lines are dried; this process may take 30 – 90 minutes depending on how dry the carrier gas supply is.

(15) When background drift is relatively stable, and preferably below 20 microgram per minute, the unit is ready for sample analysis.

4.4 Programming the Aquamax KF Coulometric

Note: Refer to the Aquamax KF Coulometer user manual.

Programme the titrator to provide results in ppm or % as required, and select calculation mode W/K.

$$\text{Result (ppm or \%)} = \frac{\text{microgram count - bl}}{W} \times K$$

Where:

W	=	weight of sample dissolved (approx. 5.0 g)	
K	=	total volume of diluent + sample	(10.0ml) K = 10
		volume injected into Aquamax	(1.0ml)
bl	=	micrograms of water for 1.0ml of diluent (µg)	

To allow time for the injected sample to be heated, the water liberated and carried through to the titration vessel by the gas stream, it is advisable to programme a Start Delay of, for example, 1.0 minute. It can also be helpful to programme a Minimum Titration Time of, for example, 2.0 minutes.

To conform to ASTM D6304 add 5 ± 0.01g of accurately weighed sample in a 10ml volumetric flask. Make up to volume with dried hexane. Shake the sample until it is completely dissolved in the solvent.

Inject 1ml of dissolved sample into the evaporator vessel and start the titrator.

Chapter 5 Basics of Operation

5.1 Switching ON and OFF

The Aquamax KF Oil Evaporator has one I/O switch and is powered by either a mains power supply using an adaptor, or a built-in battery for power.

Note: Refer to Chapter 4.2 for power supply connections.

To avoid risk of electrocution, make sure the I/O switch is set to OFF before connecting the power supply from a mains source.

5.2 How to Run a Test

The Aquamax KF Oil Evaporator has one I/O switch and is powered by either a mains power supply using an adaptor, or a built-in battery for power.

(1) Confirm that Aquamax KF is in “Ready” mode and background drift is stable.

(2) Flush 1.0ml syringe several times with dissolved sample.

(3) Fit luer needle and flush through with sample.

(4) Draw dissolved sample for analysis into syringe beyond the 1.0ml marking.

(5) Invert syringe so that any air bubbles can be ejected through the needle and adjust syringe plunger to the 1.0 ml mark.

(6) Wipe off excess sample from outside of needle using a clean, dry tissue or paper towel.

(7) Press titrator [START].

(8) Pierce needle through injection septa of oil evaporator vessel.

(9) Inject dissolved sample into the base oil.

(10) Withdraw syringe from evaporator vessel.

5.3 Setting the Gas Flow Rate

The gas flow meter has a needle valve which can be adjusted to change the gas flow rate. Most applications should be run at 300 ml / minute.

(1) Set the display to 0.30.

(2) Turn gas valve clock wise to decrease and anti-clock wise to increase gas flow.

(3) Use the locking ring to lock into place.

5.4 Setting the Evaporator Temperature

(1) Press [Set]. The display shows St 1 and then shows selected temperature.

(2) To change set temperature press arrows until desired temperature is shown then press [Set] to confirm new value.

(3) The display will then return to standard view.

5.5 Closing Down and Emptying the Vessel

The evaporator vessel can be used for up to 20 sample analyses before it is necessary to empty. When samples have been completed simply switch the evaporator to OFF and turn off the dry gas supply.

Disconnect the titration vessel gas inlet adaptor from the oil evaporator.

Allow the evaporator vessel to cool sufficiently before using a suitable size syringe or similar device to withdraw the base oil and sample mixture from the vessel.

Do not switch on the heater until the base oil has been replaced.

Chapter 6 Technical Specifications

Model:	510000
Heater Type:	Silicon Mat Heater
Temperature Range:	Ambient to 200°C
Temperature Control:	Digital Setting
Gas Flow Rate:	300ml / Minute
Gas Flow Control:	Digital Setting
Carrier Gas:	Nitrogen Gas (Not Supplied)
Desiccant:	Molecular Siev
Power Supply:	24v DC 100 – 240v AC 50 – 60 Hz
Dimensions:	180 x 240 x 210 mm
Weight:	3kg

Chapter 7 Parts and Accessories

Part No.	Product
510000	Aquamax KF Oil Evaporator
All of the below is supplied with the instrument but are also available as spare items.	
401528	Power Adaptor
503086	Glass Vessel
503087	Vessel Lid
503088	Red Compression Fitting (4pk)
503089	Injection Septa (20pk)
503091	Vessel Gas Distribution Tube
503092	Pack 1 Large & 3 Small “O” Ring
503093	Gas Inlet Tube with 3-way Cock
503094	Gas Outlet Tube
503095	Desiccant Bottle
503096	Bottle Molecular Sieve
503097	Thermocouple K Type
503098	Plastic Tubes (4pk)
503099	Base Oil, 1 litre
503100	Silicone Grease (tube)

Chapter 8 Conformity and Warranty

8.1 Declaration of Conformity

GR Scientific products are in conformity with the EU Directives:

- 2004/108/EC Electromagnetic Compatibility Directive
- 2006/95/EC Low Voltage Directive

This is based upon the compliance of the products with the harmonised standards:

- BS EN 61000-4-2:2009 Electromagnetic Compatibility. Generic immunity standard. Residential, commercial and light industry;
- BS EN 61010-2-030:2010 Safety requirements for electrical equipment for measurement, control and laboratory use. Particular requirements for testing and measuring circuits;
- BS EN 61326-1:2013 Electrical equipment for measurement, control and laboratory use. EMC requirements. General requirements.

8.2 Quality Control

The Aquamax KF Coulometric; is adequately and professionally Quality Controlled to ensure that it is fully functional and to ensure that it meets all of the requirements that the instrument supports.

8.3 Customer Support and Service

We want you to maximise the return on your equipment through a service program tailored to your requirements from when you purchase our equipment which includes:

- Support and Repair
- Maintenance and Calibration
- Training and Installation
- Quality Assurance and Certification

Our dedicated service staff are on hand to help in the unlikely event that your equipment develops a fault. Please contact us using the details provided to discuss the next step.

8.4 Warranty

Under the conditions of our warranty, all faults which are proved to be due to material, construction or manufacturing defects, which occur within 24 months of the delivery date, will be repaired or replaced, (at our discretion), free of charge at our premises. Freight costs will be chargeable along with any inspection work which was not necessitated by manufacturing or material defects. Breakages of glassware or electrodes are not covered by this guarantee. In the event of instrument failure during the warranty period, written authorisation must be obtained from the manufacturer prior to shipping.

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